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1-[2-[(2,4-Dichlorobenzylidene)amino]ethyl]-3-methylimidazolium hexafluorophosphate

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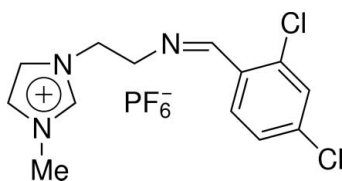
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.031; wR factor = 0.093; data-to-parameter ratio = 15.7.

In the title Schiff base compound, $\text{C}_{13}\text{H}_{14}\text{Cl}_2\text{N}_3^+\cdot\text{PF}_6^-$, the dihedral angle between the aromatic ring and imidazole ring in the cation is $6.10(2)^\circ$. Intermolecular $\text{C}-\text{H}\cdots\text{F}$ hydrogen-bonding interactions and $\pi-\pi$ stacking interactions [centroid-centroid distance = $3.7203(12)$ Å] help stabilize the crystal packing.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related structures, see: Pradeep (2005); Li *et al.* (2009). For ionic liquids and their applications, see: Wasserscheid & Keim (2000); Singh & Sekhon (2005); Noda & Watanabe (2000).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{14}\text{Cl}_2\text{N}_3^+\cdot\text{PF}_6^-$
 $M_r = 428.14$
 Triclinic, $P\bar{1}$
 $a = 8.3465(13)$ Å
 $b = 10.1419(16)$ Å
 $c = 11.0310(17)$ Å

$\alpha = 78.899(2)^\circ$
 $\beta = 76.523(2)^\circ$
 $\gamma = 67.834(2)^\circ$
 $V = 835.3(2)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.55$ mm⁻¹
 $T = 173$ K

$0.32 \times 0.24 \times 0.21$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.844$, $T_{\max} = 0.894$

3566 measured reflections
 3566 independent reflections
 3151 reflections with $I > 2\sigma(I)$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.093$
 $S = 1.10$
 3566 reflections

227 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.25$ e Å⁻³
 $\Delta\rho_{\min} = -0.34$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C5}-\text{H5}\cdots\text{F3}$	0.95	2.51	3.324 (2)	143
$\text{C10}-\text{H10}\cdots\text{F3}^i$	0.95	2.33	3.203 (2)	152
$\text{C10}-\text{H10}\cdots\text{F5}^i$	0.95	2.54	3.373 (2)	147
$\text{C11}-\text{H11}\cdots\text{F6}$	0.95	2.46	3.275 (2)	143
$\text{C12}-\text{H12}\cdots\text{F2}$	0.95	2.48	3.239 (2)	137
$\text{C13}-\text{H13C}\cdots\text{F5}^{ii}$	0.98	2.54	3.464 (2)	158

Symmetry codes: (i) $x - 1, y + 1, z$; (ii) $-x + 1, -y + 1, -z$.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2012).

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supplementary materials

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1-{2-[(2,4-Dichlorobenzylidene)amino]ethyl}-3-methylimidazolium hexafluorophosphate

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Comment

Ionic liquids are attracting much interest in many fields of chemistry and industry, due to their potential as green solvents for a wide range of applications in synthesis, catalysis, electrochemistry, and liquid-liquid extractions (Wasserscheid *et al.*, 2000; Singh, 2005; Noda, 2000). Schiff base compounds are one of most prevalent mixed-donor ligands in the field of coordination chemistry (Li *et al.*, 2009). As part of our program aimed at developing a novel functionalized ionic liquid, we now report the crystal structure of a novel ionic liquid-supported Schiff base (I).

The asymmetric unit of the title compound, (I), a Schiff base derived ionic liquid, is comprised of an organic cation and a PF₆ counter anion, Fig. 1. Bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987). The dihedral angle between the mean planes of the imidazole and benzene rings in the cation is 6.10°. The crystal structure exhibits weak C–H···Cl intramolecular and C–H···F intermolecular hydrogen bonding interactions as well as aromatic π – π stacking interactions between the imidazole and benzene rings of neighbouring cations [Cg1···Cg2 = 3.7203 (12)Å; 1-x, 1-y, 1-z, where Cg1 and Cg2 are centroids of the imidazole (N1/C10/N2/C11/C12) and benzene (C1–C6) rings, respectively, Fig. 2].

Experimental

A mixture of the ionic liquid 1-(2-aminoethyl)-3-methylimidazolium hexafluorophosphate (4 mmol) and 2,4-dichlorobenzaldehyde (3 mmol) was stirred for 4 h at room temperature under solvent-free conditions. After completion of the reaction, ethanol (30 ml) was added to the reaction mixture, filtered off the solid product and washed with cold ethanol. The crude product was purified by recrystallization in ethanol/ethyl acetate (3:1 v/v). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethyl acetate solution of the complex at room temperature.

Refinement

All H atoms were located in a difference Fourier maps and were refined as riding atoms: C–H = 0.95–0.99 Å and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Figures

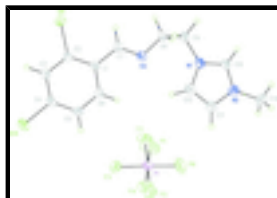


Fig. 1. The molecular structure of the title compound in (I) showing the atom numbering Scheme. Displacement ellipsoids are drawn at the 50% probability level.

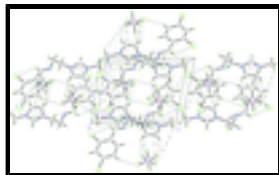


Fig. 2. Packing diagram of (I) viewed down the b axis. Weak C–H...Cl intramolecular and C–H...F intermolecular hydrogen bonding interactions are shown as dashed lines.

1-{2-[(2,4-Dichlorobenzylidene)amino]ethyl}-3-methylimidazolium hexafluorophosphate

Crystal data

$C_{13}H_{14}Cl_2N_3^+ \cdot P_1F_6^-$	$Z = 2$
$M_r = 428.14$	$F_{000} = 432$
Triclinic, $P\bar{1}$	$D_x = 1.702 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 8.3465 (13) \text{ \AA}$	Cell parameters from 5376 reflections
$b = 10.1419 (16) \text{ \AA}$	$\theta = 2.7\text{--}27.0^\circ$
$c = 11.0310 (17) \text{ \AA}$	$\mu = 0.55 \text{ mm}^{-1}$
$\alpha = 78.899 (2)^\circ$	$T = 173 \text{ K}$
$\beta = 76.523 (2)^\circ$	Block, colorless
$\gamma = 67.834 (2)^\circ$	$0.32 \times 0.24 \times 0.21 \text{ mm}$
$V = 835.3 (2) \text{ \AA}^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	3566 independent reflections
Radiation source: fine-focus sealed tube	3151 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.0000$
$T = 173 \text{ K}$	$\theta_{\text{max}} = 27.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.844$, $T_{\text{max}} = 0.894$	$k = -12 \rightarrow 12$
3566 measured reflections	$l = -14 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.3955P]$
$S = 1.10$	where $P = (F_o^2 + 2F_c^2)/3$
3566 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
227 parameters	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$

Primary atom site location: structure-invariant direct methods Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3066 (2)	0.34376 (17)	0.75848 (15)	0.0220 (3)
C2	0.3555 (2)	0.30042 (17)	0.87666 (15)	0.0227 (3)
C3	0.4986 (2)	0.17904 (17)	0.90058 (15)	0.0241 (3)
H3	0.5296	0.1513	0.9817	0.029*
C4	0.5944 (2)	0.09983 (17)	0.80205 (16)	0.0240 (3)
C5	0.5499 (2)	0.13684 (18)	0.68374 (16)	0.0270 (3)
H5	0.6167	0.0796	0.6180	0.032*
C6	0.4071 (2)	0.25808 (18)	0.66287 (15)	0.0257 (3)
H6	0.3761	0.2841	0.5818	0.031*
C7	0.1562 (2)	0.47512 (17)	0.73301 (15)	0.0237 (3)
H7	0.0901	0.5323	0.7989	0.028*
C8	-0.0351 (2)	0.64629 (18)	0.61084 (16)	0.0283 (4)
H8A	-0.0911	0.6807	0.6939	0.034*
H8B	-0.1235	0.6282	0.5765	0.034*
C9	0.0251 (2)	0.76060 (18)	0.52316 (16)	0.0297 (4)
H9A	-0.0739	0.8533	0.5247	0.036*
H9B	0.1211	0.7719	0.5539	0.036*
C10	0.0061 (2)	0.79621 (17)	0.29722 (15)	0.0244 (3)
H10	-0.1003	0.8765	0.3020	0.029*
C11	0.2433 (2)	0.62263 (18)	0.22472 (17)	0.0295 (4)
H11	0.3313	0.5609	0.1688	0.035*
C12	0.2369 (2)	0.61583 (18)	0.34869 (17)	0.0285 (4)
H12	0.3197	0.5483	0.3968	0.034*
C13	0.0537 (3)	0.7880 (2)	0.06825 (17)	0.0368 (4)
H13A	-0.0631	0.8627	0.0741	0.055*
H13B	0.0539	0.7081	0.0304	0.055*
H13C	0.1410	0.8276	0.0161	0.055*
Cl1	0.23667 (6)	0.39837 (5)	1.00213 (4)	0.03149 (12)
Cl2	0.78059 (6)	-0.04833 (5)	0.82692 (4)	0.03387 (13)
F1	0.44475 (17)	0.19348 (17)	0.31806 (17)	0.0655 (4)
F2	0.58081 (16)	0.32801 (12)	0.36186 (12)	0.0436 (3)

supplementary materials

F3	0.70635 (18)	0.08681 (13)	0.38373 (11)	0.0492 (3)
F4	0.82315 (14)	0.21845 (14)	0.22679 (11)	0.0412 (3)
F5	0.68843 (16)	0.08228 (12)	0.18313 (11)	0.0423 (3)
F6	0.56548 (19)	0.32368 (14)	0.15939 (13)	0.0555 (4)
N1	0.08759 (18)	0.72527 (14)	0.39294 (13)	0.0243 (3)
N2	0.09752 (19)	0.73655 (15)	0.19419 (13)	0.0262 (3)
N3	0.11363 (19)	0.51306 (15)	0.62537 (13)	0.0263 (3)
P1	0.63280 (5)	0.20636 (5)	0.27210 (4)	0.02671 (12)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0223 (7)	0.0219 (8)	0.0212 (7)	-0.0075 (6)	-0.0039 (6)	-0.0016 (6)
C2	0.0239 (7)	0.0235 (8)	0.0204 (7)	-0.0080 (6)	-0.0019 (6)	-0.0049 (6)
C3	0.0259 (8)	0.0241 (8)	0.0227 (8)	-0.0088 (6)	-0.0070 (6)	-0.0002 (6)
C4	0.0216 (7)	0.0178 (7)	0.0298 (8)	-0.0042 (6)	-0.0047 (6)	-0.0016 (6)
C5	0.0284 (8)	0.0242 (8)	0.0259 (8)	-0.0062 (7)	-0.0016 (6)	-0.0070 (6)
C6	0.0280 (8)	0.0272 (8)	0.0204 (7)	-0.0075 (7)	-0.0048 (6)	-0.0027 (6)
C7	0.0235 (8)	0.0223 (8)	0.0233 (8)	-0.0057 (6)	-0.0038 (6)	-0.0031 (6)
C8	0.0278 (8)	0.0279 (9)	0.0233 (8)	-0.0011 (7)	-0.0079 (6)	-0.0032 (6)
C9	0.0379 (9)	0.0236 (8)	0.0257 (8)	-0.0038 (7)	-0.0120 (7)	-0.0050 (6)
C10	0.0256 (8)	0.0197 (7)	0.0260 (8)	-0.0053 (6)	-0.0062 (6)	-0.0019 (6)
C11	0.0259 (8)	0.0229 (8)	0.0361 (9)	-0.0073 (7)	0.0005 (7)	-0.0053 (7)
C12	0.0217 (8)	0.0223 (8)	0.0373 (9)	-0.0034 (6)	-0.0060 (7)	-0.0013 (7)
C13	0.0491 (11)	0.0397 (10)	0.0220 (8)	-0.0165 (9)	-0.0065 (8)	-0.0017 (7)
Cl1	0.0345 (2)	0.0313 (2)	0.0223 (2)	-0.00086 (17)	-0.00639 (16)	-0.00879 (16)
Cl2	0.0295 (2)	0.0252 (2)	0.0392 (2)	0.00147 (16)	-0.00988 (17)	-0.00417 (17)
F1	0.0325 (7)	0.0723 (10)	0.0967 (12)	-0.0247 (7)	0.0080 (7)	-0.0315 (9)
F2	0.0411 (6)	0.0347 (6)	0.0488 (7)	-0.0033 (5)	-0.0004 (5)	-0.0202 (5)
F3	0.0676 (9)	0.0348 (6)	0.0269 (6)	-0.0002 (6)	-0.0056 (5)	-0.0010 (5)
F4	0.0279 (6)	0.0566 (7)	0.0404 (6)	-0.0150 (5)	0.0001 (5)	-0.0158 (5)
F5	0.0519 (7)	0.0373 (6)	0.0390 (6)	-0.0120 (5)	-0.0087 (5)	-0.0140 (5)
F6	0.0626 (9)	0.0399 (7)	0.0548 (8)	0.0017 (6)	-0.0332 (7)	0.0033 (6)
N1	0.0253 (7)	0.0194 (6)	0.0269 (7)	-0.0046 (5)	-0.0080 (5)	-0.0027 (5)
N2	0.0282 (7)	0.0245 (7)	0.0249 (7)	-0.0092 (6)	-0.0028 (5)	-0.0028 (5)
N3	0.0277 (7)	0.0235 (7)	0.0244 (7)	-0.0038 (6)	-0.0072 (5)	-0.0024 (5)
P1	0.0213 (2)	0.0254 (2)	0.0292 (2)	-0.00174 (17)	-0.00511 (16)	-0.00594 (17)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.397 (2)	C9—H9B	0.9900
C1—C6	1.401 (2)	C10—N2	1.324 (2)
C1—C7	1.479 (2)	C10—N1	1.330 (2)
C2—C3	1.387 (2)	C10—H10	0.9500
C2—Cl1	1.7414 (16)	C11—C12	1.345 (3)
C3—C4	1.383 (2)	C11—N2	1.382 (2)
C3—H3	0.9500	C11—H11	0.9500
C4—C5	1.383 (2)	C12—N1	1.380 (2)
C4—Cl2	1.7394 (16)	C12—H12	0.9500

C5—C6	1.378 (2)	C13—N2	1.466 (2)
C5—H5	0.9500	C13—H13A	0.9800
C6—H6	0.9500	C13—H13B	0.9800
C7—N3	1.266 (2)	C13—H13C	0.9800
C7—H7	0.9500	F1—P1	1.5798 (13)
C8—N3	1.462 (2)	F2—P1	1.5991 (12)
C8—C9	1.521 (3)	F3—P1	1.6059 (12)
C8—H8A	0.9900	F4—P1	1.5945 (12)
C8—H8B	0.9900	F5—P1	1.6077 (12)
C9—N1	1.474 (2)	F6—P1	1.5903 (13)
C9—H9A	0.9900		
C2—C1—C6	117.32 (15)	N1—C10—H10	125.6
C2—C1—C7	122.19 (14)	C12—C11—N2	106.95 (15)
C6—C1—C7	120.49 (14)	C12—C11—H11	126.5
C3—C2—C1	122.51 (15)	N2—C11—H11	126.5
C3—C2—C11	116.75 (12)	C11—C12—N1	107.24 (15)
C1—C2—C11	120.74 (12)	C11—C12—H12	126.4
C4—C3—C2	117.56 (15)	N1—C12—H12	126.4
C4—C3—H3	121.2	N2—C13—H13A	109.5
C2—C3—H3	121.2	N2—C13—H13B	109.5
C3—C4—C5	122.21 (15)	H13A—C13—H13B	109.5
C3—C4—C12	118.75 (13)	N2—C13—H13C	109.5
C5—C4—C12	119.02 (13)	H13A—C13—H13C	109.5
C6—C5—C4	118.87 (15)	H13B—C13—H13C	109.5
C6—C5—H5	120.6	C10—N1—C12	108.39 (14)
C4—C5—H5	120.6	C10—N1—C9	124.53 (14)
C5—C6—C1	121.51 (15)	C12—N1—C9	127.08 (14)
C5—C6—H6	119.2	C10—N2—C11	108.67 (14)
C1—C6—H6	119.2	C10—N2—C13	125.07 (15)
N3—C7—C1	121.45 (15)	C11—N2—C13	126.20 (15)
N3—C7—H7	119.3	C7—N3—C8	116.53 (14)
C1—C7—H7	119.3	F1—P1—F6	91.02 (9)
N3—C8—C9	110.67 (14)	F1—P1—F4	179.50 (9)
N3—C8—H8A	109.5	F6—P1—F4	89.48 (8)
C9—C8—H8A	109.5	F1—P1—F2	90.12 (7)
N3—C8—H8B	109.5	F6—P1—F2	91.08 (7)
C9—C8—H8B	109.5	F4—P1—F2	89.81 (6)
H8A—C8—H8B	108.1	F1—P1—F3	90.46 (9)
N1—C9—C8	112.47 (14)	F6—P1—F3	178.38 (8)
N1—C9—H9A	109.1	F4—P1—F3	89.04 (7)
C8—C9—H9A	109.1	F2—P1—F3	89.56 (7)
N1—C9—H9B	109.1	F1—P1—F5	90.54 (7)
C8—C9—H9B	109.1	F6—P1—F5	89.83 (7)
H9A—C9—H9B	107.8	F4—P1—F5	89.52 (6)
N2—C10—N1	108.75 (14)	F2—P1—F5	178.86 (7)
N2—C10—H10	125.6	F3—P1—F5	89.51 (7)
C6—C1—C2—C3	0.9 (2)	N3—C8—C9—N1	67.10 (18)
C7—C1—C2—C3	-178.59 (15)	N2—C11—C12—N1	0.05 (19)

supplementary materials

C6—C1—C2—C11	-179.03 (12)	N2—C10—N1—C12	-0.11 (18)
C7—C1—C2—C11	1.5 (2)	N2—C10—N1—C9	179.91 (15)
C1—C2—C3—C4	0.0 (2)	C11—C12—N1—C10	0.03 (19)
C11—C2—C3—C4	179.95 (12)	C11—C12—N1—C9	-179.99 (16)
C2—C3—C4—C5	-1.1 (2)	C8—C9—N1—C10	111.90 (18)
C2—C3—C4—C12	177.09 (12)	C8—C9—N1—C12	-68.1 (2)
C3—C4—C5—C6	1.1 (3)	N1—C10—N2—C11	0.14 (18)
C12—C4—C5—C6	-177.02 (13)	N1—C10—N2—C13	-177.43 (15)
C4—C5—C6—C1	-0.1 (3)	C12—C11—N2—C10	-0.12 (19)
C2—C1—C6—C5	-0.8 (2)	C12—C11—N2—C13	177.41 (16)
C7—C1—C6—C5	178.67 (15)	C1—C7—N3—C8	-178.82 (14)
C2—C1—C7—N3	179.95 (16)	C9—C8—N3—C7	113.18 (17)
C6—C1—C7—N3	0.5 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C7—H7 \cdots C11	0.95	2.69	3.0846 (17)	106
C12—H12 \cdots F2	0.95	2.48	3.239 (2)	137
C5—H5 \cdots F3	0.95	2.51	3.324 (2)	143
C11—H11 \cdots F6	0.95	2.46	3.275 (2)	143
C10—H10 \cdots F3 ⁱ	0.95	2.33	3.203 (2)	152
C10—H10 \cdots F5 ⁱ	0.95	2.54	3.373 (2)	147
C13—H13C \cdots F5 ⁱⁱ	0.98	2.54	3.464 (2)	158

Symmetry codes: (i) $x-1, y+1, z$; (ii) $-x+1, -y+1, -z$.

Fig. 2

